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(54) Title: PROCESS FOR ENHANCING THE CHARACTERISTICS AND DURABILITY OF WOOD FIBER CEMENT AND WOOD FIBER CONCRETE COMPOSITES

(57) Abstract: The invention relates to materials, particularly composite materials and enhancement of their properties. The invention provides a method for enhancing the properties of wood fiber cement and wood fiber concrete composites comprising treating the wood fibers with an aqueous chemical before combining with cement or concrete. The aqueous chemical is preferably acrylic emulsion or alkylalkoxysilane. The treatment enhances the dimensional stability and compressive strentgh of the composites. The invention also provides the resulting composites. These enhanced composites are suitablefor use as building materials.

TITLE: PROCESS FOR ENHANCING THE CHARACTERISTICS AND DURABILITY OF WOOD FIBER CEMENT AND WOOD FIBER CONCRETE COMPOSITES

CROSS-REFERENCE TO RELATED APPLICATION

This application claims the benefit of U.S. Provisional Patent Application No. 60/135,016, filed May 20, 1999.

TECHNICAL FIELD

This invention relates to the field of materials, particularly composite materials. Most particularly, the invention relates to the enhancement of characteristics of wood fiber cement or wood fiber concrete composites.

BACKGROUND OF THE INVENTION

Portland and other inorganic cements are combined with different aggregates and/or fibers to produce a wide variety of concretes. These composites constitute the largest annual consumption of manmade materials in the world. These cementious composites have reasonable mechanical properties but are considered to be brittle, weak in tension, and low in toughness.

The idea of using plant fibers, including wood fiber, as reinforcement for cementious materials dates back as far as biblical times. In this context a cementious composite is defined as a matrix of cement containing reinforcing fibers. The idea was first recorded around 3500 years ago when straw was added to reinforce sun-baked bricks, and then around 1900 asbestos was used to reinforce cement.

Nearly half a century ago in Europe, the first wood cement composite produced was excelsior board, which was later modified to add Portland cement as a binder. Wood fiber reinforced cement complements the high tensile strength, toughness, impact resistance, and workability of

wood with the fire resistance, durability and dimensional stability of cement-based materials. These boards were produced in the U.S. and used for roof decking and wall paneling. Products comprised of excelsior have good acoustic and fire resistance, but only moderate strength and thermal insulation values. Out of these capabilities came cement-bonded wood fiber composites. A high-density cement-bonded wood flakeboard or particleboard was later investigated which in turn led to further applications.

Wood cement panels, roof shingles, and other building products are widely accepted in Europe and Japan because of certain desirable characteristics, such as high fire, insect, and fungal resistance as well as good weatherability and acoustic insulation properties. Use of wood cement composites in the U.S. lags Europe and Japan but is gaining acceptance in certain applications such as roof shingles and as ceramic tile backing panels. U.S. consumption of wood cement composites as a building material is increasing annually and the use of recycled wood fibers in these composites is on the rise.

The properties of wood cement composites need to be enhanced if the uses of wood fiber cement composites are going to increase and the applications as a building material are also going to expand. Limiting properties for the use of any material include mechanical properties, dimensional stability, and durability. Cement is a brittle material, and wood is a viscoelastic material. Enhancing the properties of wood fiber cement composites means improving the properties of the cement by transferring as much as possible the elastic properties of the wood to the composite. Attempts have been made to do this by just mixing wood and cement and by adding chemical additives to the cement water mix prior to mixing in the wood fibers. These attempts resulted in only slight improvements in the composite properties.

For the foregoing reasons, a need exists for methods to more significantly improve the properties of wood fiber cement and wood fiber concrete composites.

SUMMARY OF THE INVENTION

An object of the invention is a method for improving the properties of wood fiber cement and wood fiber concrete composites by pretreating the wood fibers.

Another object of the invention is to provide wood fiber cement and wood fiber concrete composites with improved properties, such as improved dimensional stability and improved durability.

These and other objects, features, and advantages will become apparent after review of the following description and claims of the invention which follow.

The present invention relates to treating wood fibers with aqueous solutions prior to fabrication into composites in order to enhance the properties of wood fiber cement or concrete composites. Any water soluble organic or inorganic chemical that is compatible with wood fibers and cement-based binders can be used to enhance the properties of the composite compared to using untreated wood fibers. Treated and untreated wood fiber has also been used to reinforce concrete.

The aqueous solutions of inorganic or organic compounds modify the surfaces of the wood fiber improving the coupling or transfer of properties between the wood fiber and the cement.

The pre-treatment of the fibers with aqueous solutions improved the characteristics of the wood fiber cement and wood fiber concrete composites as evidenced by behavior during moisture cycling and temperature cycling. The result of the present wood fiber surface treatment was significant improvement in the durability, toughness, and dimensional stability of the treated wood fiber cement

composites compared to untreated wood fiber cement composites.

Enhanced wood fiber cement composites will expand their application and use as a building material in construction. The technology will also expand the use of recycled wood fiber in concrete.

DETAILED DESCRIPTION OF THE INVENTION

Wood fibers, including secondary wood from recycled paper, may be used as a reinforcement in cement. Processing secondary wood fibers into fibrous form is helpful in dispersing the wood fiber in the cement matrix. Enhanced wood fiber cement composite properties may be obtained by treating the wood fibers with aqueous chemicals prior to fabrication into the composites. The effects of long-term moisture cycling on dimensional stability and long-term temperature cycling on the compressive strength of wood fiber cement composites was explored.

Moisture cycling effects on the dimensional stability of wood fiber cement composites were investigated. The study used Kraft and hardwood fibers treated with an aqueous acrylic emulsion or aqueous alkylalkoxysilane prior to manufacturing the wood fiber cement composites. The treated fiber cement composites were more resistant to moisture cycling deterioration than the neat cement and the untreated fiber cement composites. The alkylalkoxysilane treated fibers were more resistant to moisture cycling deterioration than the acrylic emulsion treated fiber cement composites. The treated hardwood fibers had more moisture resistance than the treated Kraft fiber composites within each treatment.

The effects of temperature cycling on the average compressive strength values produced somewhat similar results to the moisture cycling. The acrylic emulsion and the alkylalkoxysilane treated fiber composites resisted

deterioration compared to the neat cement specimens. The alkylalkoxysilane treated fibers had higher average compressive strength values compared to the neat and acrylic emulsion treated specimens.

The specific aqueous chemical treatments tested are described in detail below. Though treatment with any water soluble organic or inorganic chemical compatible with the wood fibers and cement-based binders is believed to enhance the properties of the composites, the preferred chemicals are aqueous acrylic emulsion and alkylalkoxysilane. The most preferred chemical is alkylalkoxysilane. Other aqueous chemicals, such as silicates, have been used to treat the wood fibers and enhance the resultant properties of the composites filled with the wood fibers. One of ordinary skill in the art would be able to determine additional chemicals that will work with the invention.

The wood fibers used in the invention are not critical. Bleached hardwood pulp and unbleached recycled Kraft paper were used. One of ordinary skill in the art would be able to determine wood fibers that will work with the invention.

The cement or concrete is also not critical. Type III Portland cement was used. One of ordinary skill in the art would be able to determine cement or concrete that will work with the invention.

The examples below demonstrate the preferred embodiment of the invention. One of ordinary skill in the art would be able to readily determine ranges of ratios of ingredients, concentrations of aqueous chemical, treatment time, and other variations on the methods and compositions taught. For example, a 10% by volume solution of the aqueous chemical was used. One of ordinary skill in the art would be able to determine concentrations of chemicals that will work with the invention.

EXAMPLES

Materials

Wood fibers used in the study were bleached hardwood pulp and unbleached recycled Kraft paper. Kraft paper is composed of softwood fibers.

The cement used in the study was ASTM Type III Portland cement obtained from Lehigh Portland Cement Company. The composition of the cement is given Table 1.

Table 1. Chemical composition of the cement used in the study.

Chemical	ASTM Type III Portland Cement (Oxide Equivalent Weight %)
CaO	63.70
SiO_2	20.70
Al_2O_3	4.20
MgO	3.70
Fe_2O_3	2.30
SO_3	3.10
Na_2O equiv.	0.57
LOI	2.00
Insoluble Residue	0.20

Preparation of fibers

Recycled paper fiber or pulp was soaked in de-ionized water at room temperature for more than ½ hour before mechanical beating in water using a Valley Beater. The wood fibers were then removed from the slurry by vacuum filtration. The fibers were separated by milling after air drying. The wood fibers were passed through a 2mm screen after milling.

The pulp fibers that were treated with the aqueous chemicals were placed in the aqueous solution (by volume) of diluted chemicals for 30 minutes. After treating the surface of the wood fibers, the fibers were air-dried and milled so that they passed a 2mm screen.

Wood fiber cement specimen preparation

The pulp fibers and treated pulp fiber composites used in the study were as follows:

1. Neat cement was Type III Portland Cement.
2. Untreated hardwood pulp mixed with Type III Portland Cement.
3. Untreated Kraft pulp mixed with Type III Portland Cement.
4. A60-Kraft Pulp was 10% aqueous solution (by volume) of Acryl-60 (acrylic emulsion) treated Kraft pulp fiber mixed with Type III Portland Cement.
5. A60-Hardwood Pulp was 10% aqueous solution (by volume) of Acryl-60 (acrylic emulsion) treated Hardwood pulp fiber mixed with Type III Portland Cement.
6. Si-Kraft Pulp was 10% aqueous solution (by volume) of Environseal 20 (solution of 20% alkylalkoxysilane in water) treated Kraft pulp fiber mixed with Type III Portland Cement.
7. Si-Hardwood Pulp was 10% aqueous solution (by volume) of Environseal 20 (solution of 20% alkylalkoxysilane in water) treated Hardwood pulp fiber mixed with Type III Portland Cement.

The basic formulations for the specimens in the study are given in Table 2.

Table 2. Basic specimen formulations.

Sample	Weight % of components			
	Cement	Deionized water	Fibers	Super-plasticizer
Control*	75.99	21.25	0.00	2.76
WFRC+	67.38	25.13	4.24	3.25

*W/C = 0.28

+W/C = 0.37

The wood fiber composites were mixed using a paddle style mixer. The dry wood fibers and cement with superplasticizer (Borem 100 HMP, supplied by Boremco Specialty Chemicals) were mixed in a plastic bag. The dry mixture of wood fibers and cement was placed into a bowl containing the water for ½ minute. The paste was mixed at slow speed for ½ minute. After scraping down the sides of the bowl and the mixing paddle, the paste was mixed at medium speed for another 2½ minutes.

Samples were molded in the size required for testing (e.g., 25.4 by 25.4 by 127 mm bars for dimensional stability, and 50.8 mm cubes for compression specimens), demolded after one day of curing, and continuously cured (at ~100% relative humidity, ambient pressure and temperature 25°C for 28 days) prior to temperature cycling and testing.

Property measurements

The effects of moisture cycling on dimensional stability included measuring the length, width, thickness, and weight of 3 specimens at each treatment level after each wet or dry cycle. The wet/dry cycling started from the wet condition (7 days in 25°C water) to the dry condition (7 days in 40°C, 29-34% relative humidity). The

dimensional stability property measurements were obtained every 7 days until each specimen broke due to expansion and contraction.

The effects of temperature cycling on the compressive strength of 3 specimens were measured for each treatment level at selected temperature cycle intervals. The compressive test specimens were submerged in water and placed in a temperature-controlled chamber. The chamber used for the temperature cycling was heated and cooled from approximately 1°C to -1°C to 1°C over approximately 5 hours. The compression testing was conducted at intervals of 36 cycles up to 252 cycles. At the appropriate interval, three samples of each treatment were removed and tested according to ASTM C 109 (Compressive Strength of Hydraulic Cement Mortars). A total of 105 samples (7 cycle intervals, 5 treatments and 3 replications) were prepared and tested to failure.

Example 1

Effects of Moisture Cycling

Neat cement specimens failed after 10 cycles, Kraft and hardwood fiber cement composites failed after 22 cycles and newspaper fiber cement composites failed after 25 cycles (Table 3).

Table 3. Summary of Moisture Effects from Wet-Dry Cycling on the Dimensional Properties of Wood Fiber Cement Composites.

Wood Fiber Cement Composites	Average Number Of Wet-Dry Cycles at Failure	Avg. % Change at Failure			
		Dry Cycle		Wet Cycle	
		Length	Volume	Length	Volume
Controls[1]	10	0.88	1.87	0.93	2.71
UNT K[2]	22	0.41	NA[8]	0.69	NA
UNT H[3]	22	0.15	NA	0.39	NA
A60-K[4]	29	0.01	0.24	0.08	0.48
A60-H[5]	44	0.62	0.69	0.55	1.58
Si-K[6]	50	0.09	0.27	-0.13	-0.36
Si-H[7]	55	-0.07	-0.33	-0.17	-0.30

[1] Type III Portland Cement Controls

[2] UNT K is untreated Kraft fiber and Type III Portland Cement

[3] UNT H is untreated Hardwood fiber and Type III Portland Cement

[4] A60-K is 10% (by volume) Acryl 60 (acrylic emulsion) treated Kraft fiber and Type III Portland Cement

[5] A60-H is 10% (by volume) Acryl 60 (acrylic emulsion) treated Hardwood fiber and Type III Portland Cement

[6] Si-K is 10% (by volume) alkylalkoxysilane treated Kraft fiber and Type III Portland Cement

[7] Si-H is 10% (by volume) alkylalkoxysilane treated Hardwood fiber and Type III Portland Cement

[8] NA means data not available

The effect of reinforcement by the wood fiber cement matrix compared to neat cement specimens might be attributed to: 1) the flexible and conformable structure of wood fibers allowing the fibers to accommodate a certain amount of volume change, which reduced the stress on the cement matrix and, 2) successful bridging role as the wood fiber restricts the further extension of a crack in the cement matrix under stress from wet-dry cycles.

The study was designed to determine if additional enhancement of the dimensional properties could be achieved by modifying the surface of the Kraft and hardwood fibers. Wood fiber cement composite specimens were prepared for wet-dry cycling using Type III Portland Cement as controls and wood fiber cement composite specimens using hardwood fibers treated with 10% (by volume) aqueous acrylic emulsion, hardwood fibers treated with 10% (by volume) aqueous alkylalkoxysilane, Kraft fibers treated with 10% (by volume) aqueous acrylic emulsion, and Kraft fibers treated with 10% (by volume) aqueous alkylalkoxysilane in a matrix of Type III Portland Cement.

Table 3 (above) lists a summary of moisture cycling data for the controls (neat cement) and the acrylic and silane treated Kraft and hardwood fiber cement composites. Percent change in the length and volume along with the average number of wet-dry cycles at failure is given.

Analysis of variance statistical techniques were used to determine if the treatments had a significant effect on the average number of cycles at failure. The level of significance was set at 0.025. The average number of cycles at failure for all of the treatments were significantly different from each other except for the untreated Kraft compared to the untreated hardwood cement composites.

Comparing the average percent change in length at failure for both fiber treatments using the aqueous acrylic

emulsion and the aqueous alkylalkoxysilane indicated that the dimensional properties of the wood fiber cement composites improved relative to the untreated wood fiber cement composites and the controls. The average percent linear dimension change at failure was lower for the treated wood fibers at failure than the untreated wood fiber composites and the average control value. This indicated that the treated wood fibers stabilized the dimension changes in the wood fiber cement composites.

The Kraft and hardwood fibers treated with aqueous acrylic emulsion produced similar average percent linear expansion data at failure to the untreated Kraft and hardwood fiber composite values. However, the average number of cycles at failure increased from 22 for the untreated wood fiber composites to 34 and 44 for the aqueous acrylic emulsion treated Kraft and hardwood fiber cement composites, respectively. The acrylic treatment of the wood fibers enhanced the ability of the composite to withstand wet-dry cycling.

The aqueous alkylalkoxysilane treatment of the Kraft and hardwood fibers produced enhanced results over the untreated fiber and the aqueous acrylic treated fiber composites. The average percent length change at failure was lowest for the alkylalkoxysilane treated Kraft and hardwood fiber cement composites compared to the controls, untreated Kraft and hardwood fiber cement composites, and the acrylic Kraft and hardwood cement composites. In addition, the average number of cycles at failure was the highest for the alkylalkoxysilane treated fibers. This indicated that the alkylalkoxysilane treatment was the most effective treatment used in the study for reinforcement of the cement matrix.

Example 2

Effects of Temperature Cycling

The effects of temperature cycling through freezing and thawing are presented in Table 4.

Table 4. Summary of the Average Compressive Strength Values for Wood Cement Composites After Thawing-Freezing-Thawing Temperature Cycling.

Wood Fiber Cement Specimens	Average Compressive Strength (MPa)						
	36 cycles	72 cycles	108 cycles	144 cycles	180 cycles	216 cycles	252 cycles
Controls[1]	45.4	52.9	50.0	42.6	24.4	27.5	22.7
A60-K[2]	49.0	47.3	53.0	41.4	45.8	36.7	41.1
A60-H[3]	42.3	44.4	44.3	48.3	52.0	50.2	42.2
Si-I[4]	55.0	52.9	51.1	57.1	52.2	52.9	54.2
Si-H[5]	52.5	52.4	54.1	53.0	57.0	53.5	53.5

[1] Type III Portland Cement

[2] A60-K is an aqueous solution of 10% (by volume) Acryl 60 (acrylic emulsion) treated Kraft fiber and Type III Portland Cement Composites

[3] A60-H is an aqueous solution of 10% (by volume) Acryl 60 (acrylic emulsion) treated Hardwood fiber and Type III Portland cement

[4] Si-K is an aqueous solution of 10% (by volume) alkylalkoxysilane treated Kraft fiber and Type III Portland Cement

[5] Si-H is an aqueous solution of 10% (by volume) alkylalkoxysilane treated Hardwood fiber and Type III Portland Cement

The purpose of this part of the study was to determine, within the relative confines of the test procedures, the effects that the treated wood fibers had on the compressive strength retention of the composites. The treated wood fibers were used because results from the moisture cycling study indicated that the treated fibers produced more durable composites than the untreated wood fibers.

Table 4 lists the average compression strength values for the controls (neat cement) and the aqueous acrylic and silane treated Kraft and hardwood fiber cement composites. The average compressive strength values for 3 specimens tested after intervals of 36 temperature cycles are listed in Table 4 for the specimen groups.

Analysis of variance statistical tests were used to determine any significant differences within a specimen treatment group for each 36 temperature cycling interval and for comparing treatments at each 36-cycle interval. The level of significance was set at 0.025.

The average compressive strength values for the neat cement specimens were not significantly different after 36, 72, 108 and 144 cycles and after 180, 216 and 252 cycles. However, the average compressive strength values for the neat cement specimens after 36, 72, 108 and 144 cycles were significantly different from the average compressive strength values after 180, 216 and 252 cycles. The Type III Portland Cement controls average compressive strength values decreased at 180 cycles. At the end of 252 cycles the average compressive strength values were about half of the average compressive strength values prior to 144 cycles. These results indicated that considerable deterioration occurred in the microstructure of the cement possibly associated with the development of microcracks and associated extension of these cracks due to the freeze-thaw cycling.

The statistical analysis of the acrylic-treated Kraft cement specimen average compressive strength values indicated that the average compressive strength value at 216 cycles was significantly different from all the other means. The average compressive strength was lower at 216 cycles than at all the other test intervals. However, the average compressive strength value at 252 cycles was not significantly different from all the other intervals, except at 216 cycles. The lack of a lower average compressive strength value at 252 cycles negates any definition of a deterioration trend in the data set. In addition, the average compressive strength value at 216 cycles was only about 11% lower than at 252 cycles.

Statistical analysis of the acrylic-treated hardwood fiber cement specimens indicated that the average compressive strength values were not significantly different at all intervals. This indicated that the specimens did not deteriorate.

The alkylalkoxysilane-treated Kraft and hardwood fiber specimens had higher average compressive strength value at all cycles compared to the acrylic-treated Kraft and hardwood cement specimens. Statistical analysis indicated that there were no significant differences at all intervals for the alkylalkoxysilane treated Kraft and hardwood fiber cement specimens.

Statistical analysis of the average compressive strength values for each of the treatments at each interval indicated that the neat specimen values were not significantly different from the other treatments at 36, 72, 108 and 144 intervals. At 180, 216 and 252 intervals the average compressive strength values were significantly different from the means for the treated fiber specimens. Additional significant differences among treatments were identified in some of the intervals, but there were no

consistent trends with any treated wood fiber cement composites across all intervals.

Comparing the treated hardwood fiber cement composite results with the treated Kraft fiber cement composite results indicated that the treated hardwood fibers might be better at retarding microcrack growth than the treated Kraft softwood fibers.

Kraft and hardwood fiber weights were constant for each test specimen. One possible reason for the hardwood fibers being more effective is that the number of hardwood fibers per unit weight is greater than the number of Kraft softwood fibers for the same unit weight. This is related to the fact that softwood fibers are longer than hardwood fibers. Hence, more hardwood fibers were available to bridge microcracks and retard crack extension than Kraft softwood fibers in the wood fiber cement composites.

Having described the invention with reference to particular compositions, theories of effectiveness, and the like, it will be apparent to those of skill in the art that it is not intended that the invention be limited by such illustrative embodiments or mechanisms, and that modifications can be made without departing from the scope or spirit of the invention, as defined by the appended claims. It is intended that all such obvious modifications and variations be included within the scope of the present invention as defined in the appended claims. The claims are meant to cover the claimed components and steps in any sequence which is effective to meet the objectives there intended, unless the context specifically indicates to the contrary.

What is claimed is:

1. A method for producing wood fiber cement or wood fiber concrete composites comprising treating wood fibers with an aqueous chemical and combining the treated wood fibers with cement or concrete in a desired ratio.

2. The method of claim 1 further comprising drying the fibers following treatment.

3. The method of claim 2 further comprising milling the dried fibers.

4. The method of claim 3 further comprising selecting fibers that pass through a 2 mm screen.

5. The method of claim 1 further comprising soaking the wood fibers in water prior to treatment.

6. The method of claim 5 further comprising beating the fibers.

7. The method of claim 6 further comprising filtering the fibers from the water.

8. The method of claim 7 further comprising drying the fibers.

9. The method of claim 8 further comprising separating the fibers.

10. The method of claim 9 further comprising selecting fibers which pass through a 2 mm screen.

11. The method of claim 1 wherein the wood fibers are treated for 30 minutes.

12. The method of claim 1 wherein the chemical is selected from the group consisting of acrylic emulsion and alkylalkoxysilane solution.

13. The method of claim 2 wherein the chemical is alkylalkoxysilane solution.

14. The method of claim 12 wherein the chemical is in a 10% by volume aqueous solution.

15. The method of claim 1 wherein the wood fibers are selected from the group consisting of bleached hardwood pulp and unbleached recycled Kraft paper.

16. The method of claim 1 wherein the cement is Type III Portland cement.

17. The method of claim 1 further comprising adding superplasticizer to the combination.

18. The method of claim 1 further comprising adding water to the combination.

19. The method of claim 1 further comprising molding the combination.

20. The method of claim 1 further comprising curing the combination.

21. A method for improving the characteristics of wood fiber cement or concrete composites comprising producing the composites according to the method of claim 1.

22. The method of claim 21 wherein the improved characteristics comprise dimensional stability and compressive strength when subjected to moisture cycling or temperature cycling.

23. A wood fiber cement or wood fiber concrete composite comprising wood fibers treated with an aqueous chemical and cement or concrete.

24. The composite of claim 23 wherein the aqueous chemical is selected from the group consisting of acrylic emulsion and alkylalkoxysilane.

25. The composite of claim 23 wherein the chemical is alkylalkoxysilane.

26. The composite of claim 23 further comprising superplasticizer.

27. The composite of claim 23 further comprising water.

28. The composite of claim 23 wherein the wood fibers are selected from the group consisting of bleached hardwood pulp and unbleached recycled Kraft paper.

29. The composite of claim 23 wherein the cement is Type III Portland Cement.

INTERNATIONAL SEARCH REPORT

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A. CLASSIFICATION OF SUBJECT MATTER
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According to International Patent Classification (IPC) or to both national classification and IPC

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Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US 5,735,094 A (ZEMBER) 07 April 1998, see Abstract.	1-29
A	US 5,741,844 A (NASS et al.) 21 April 1998, see Abstract.	1-29

☐ Further documents are listed in the continuation of Box C. ☐ See patent family annex.

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